IN RE APPLICATION OF
JEAN-JACQUES DONZÉ ET AL.
SERIAL NO. 10/537,062
FILED: JUNE 1, 2005
FOR: Mixtures of Fluorescent Whitening Agents

Group Art Unit: 1709
Examiner: HEVEY, JOHN A.

Commissioner of Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

SECOND DECLARATION UNDER RULE 132

I, Jean-Jacques Donzé, a citizen of France, residing in F-68740 Blodelsheim, France, hereby declare:

1. That I have studied Chemistry at the Université de Haute-Alsace, Mulhouse, France, and have been awarded the degree of a Doctor in 1973.

2. That I have been employed at the Textile Research Center in Mulhouse from 1975 to 1987.

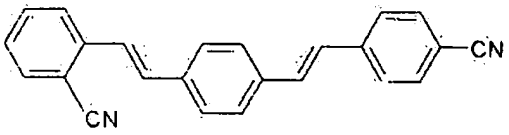
3. That I have been employed by Ciba-Geigy AG Basel, Switzerland, in January 1988, later Ciba Specialty Chemicals Inc. and Huntsman, as Chemist in the marketing of textile effects and since June 2007 in Research and Application Technology of textile effects at Huntsman.

4. That I have been engaged in application technology of fluorescent whitening agents.

5. That I am a co-inventor of U.S. Patent Application Serial No. 10/537,062 to Jean-Jacques Donzé et al. and that I am familiar with the subject matter thereof.

5. That the experiments described in the following have been made under my supervision.

Tested Fluorescent Whitening Agents (FWA):



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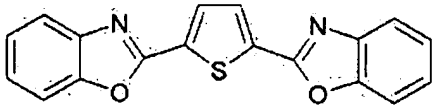


(2c)

Testing Conditions:

A polyester fabric (prescoured, heat-set at 195°C, 165 g/m^2) is treated in a dyeing apparatus at room temperature and at a liquor ratio of 1:20 with an aqueous bath containing a mixture of the fluorescent whitening agents of formula (1a) and (2c) in the ratios given in Table 1 and in the presence of 1 g/l of a fatty alcohol polyglycol ether as dispersing agent. The temperature is raised from room temperature to 130°C over 30 minutes, held for a further 30 minutes at this temperature and subsequently cooled to 40°C during 15 minutes. The textile material is then rinsed for 30 seconds under running water and dried at 70°C. The Ganz whiteness, tint value and light fastness (according to ISO 105- B02) is measured. The results are summarized in Table 1.

Table 1:

Example	Mixture of FWA	Amount	Ganz Whiteness	Tint Value	Light Fastness
A[a)]	33 % (1a) 67 % (2c)	0.12 %	215	2.2	6-7
B[b)]	17 % (1a) 83 % (2c)	0.12 %	213	1.9	7
C[b)]	15% (1a) 85% (2c)	0.12 %	212	1.9	7

a) Comparison Example according to U.S. 4,778,623 (Examples 20/22).
b) Example according to the invention.

Discussion of Results:

The obtained results show that Examples B and C according to the invention clearly show several advantages in comparison to the known Example A. The Ganz whiteness is very similar in Examples A, B and C. However, Examples B and C show

a clearly less greenish effect with a tint value below 2.0. A greenish shade is seen as a clear disadvantage by textile finishers. Additionally, Examples B and C according to the invention show a clearly better light fastness. The results are surprising and could not be expected.

In terms of lightfastness, a rating of 6 corresponds to a significant fading after 220 hours exposure in the Xenotest and 90 days exposure to daylight in Basle, CH. A rating of 7 corresponds to 400 hours in the Xenotest and 170 days exposure to daylight in Basle, CH. Thus a 6-7 rating corresponds to about 310 hours Xenotest and 130 days exposure to daylight in Basle, CH which is clearly considerably worse than a rating of 7. All textile finishers consider the difference between 6-7 and 7 as meaningful and significant.

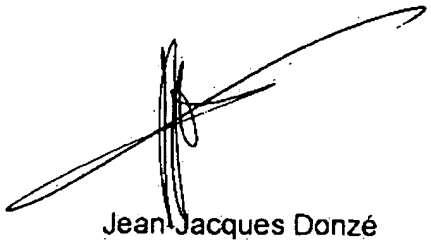
In terms of tint value for fluorescent white, the tolerance is by 0.2 difference. This tolerance is for example, specified by most brandhouses such as NIKE and Express. This tint value is an average of 4 measurements. One skilled in the art would say that the difference between 1.9 and 2.2 is known as significant when visually assessed. The whitened fabric sample with 2.2 appears really greener. It is known that such a greening is usually seen as a disadvantage by most textile finishers as being associated with a lack of development of the whitening agent.

The standard deviation for the tint value is about +/- 0.05.

There is no standard deviation for the light fastness value as a 7 verses a 6-7 value is without any tolerance or deviation due to the big difference in exposure time between the data.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed this 15th day of April, 2008



Jean Jacques Donzé

INTERNATIONAL STANDARD

ISO 105-B02

Fourth edition
1994-09-15

Textiles — Tests for colour fastness —

Part B02:
Colour fastness to artificial light: Xenon arc fading lamp test

Textiles — Essais de solidité des teintures —

Partie B02: Solidité des teintures à la lumière artificielle: Lampe à arc au xénon

Reference number
ISO 105-B02:1994(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 105-B02 was prepared by Technical Committee ISO/TC 38, *Textiles*, Subcommittee SC 1, *Tests for coloured textiles and colorants*.

This fourth edition cancels and replaces the third edition (ISO 105-B02:1988), of which it constitutes a technical revision.

ISO 105 was previously published in 13 "parts", each designated by a letter (e.g. "Part A"), with publication dates between 1978 and 1985. Each part contained a series of "sections", each designated by the respective part letter and by a two-digit serial number (e.g. "Section A01"). These sections are now being republished as separate documents, themselves designated "parts" but retaining their earlier alphanumeric designations. A complete list of these parts is given in ISO 105-A01.

Annexes A and B form an integral part of this part of ISO 105. Annex C is for information only.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland
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Textiles — Tests for colour fastness —

Part B02:
Colour fastness to artificial light: Xenon arc fading lamp test

1 Scope

This part of ISO 105 specifies a method intended for determining the resistance of the colour of textiles of all kinds and in all forms to the action of an artificial light source representative of natural daylight (D_{65}). The method is also applicable to white (bleached or optically brightened) textiles.

This method allows the use of two different sets of blue wool references. The results from the two different sets of references may not be identical.

NOTE 1 General information on colour fastness to light is given in annex C.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 105. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 105 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 105-A01:1994, *Textiles — Tests for colour fastness — Part A01: General principles of testing.*

ISO 105-A02:1993, *Textiles — Tests for colour fastness — Part A02: Grey scale for assessing change in colour.*

ISO 105-A05:—[1], *Textiles — Tests for colour fastness — Part A05: Method for the instrumental assessment of the change in colour of a test specimen.*

ISO 105-B01:—[2], *Textiles — Tests for colour fastness — Part B01: Colour fastness to light: Daylight.*

ISO 105-B05:1993, *Textiles — Tests for colour fastness — Part B05: Detection and assessment of photochromism.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

CIE Publication No. 51, *Method for assessing the quality of daylight simulators for colorimetry.*

3 Principle

A specimen of the textile to be tested is exposed to artificial light under prescribed conditions, along with a set of blue wool references. The colour fastness is assessed by comparing the change in colour of the test specimen with that of the references used.

For white (bleached or optically brightened) textiles, the colour fastness is assessed by comparing the change in whiteness of the specimens with that of the reference used.

1) To be published.

2) To be published. (Revision of ISO 105-B01:1989)

4 Reference materials and apparatus

4.1 Reference materials

Either of two sets of blue wool references may be used. The results from the two sets of references are not interchangeable.

The colour fastness ratings mentioned in this part of ISO 105 are obtained by comparison with either blue wool references 1 to 8 (preferable in Europe) or blue wool references L2 to L9 (preferable in America).

4.1.1 References 1 to 8

Blue wool references developed and produced in Europe are identified by the numerical designation 1 to 8. These references are blue wool cloths dyed with the dyes listed in table 1. They range from 1 (very low colour fastness) to 8 (very high fastness) so that each higher-numbered reference is approximately twice as fast as the preceding one.

4.1.2 References L2 to L9

Blue wool references developed and produced in the United States are identified by the letter L followed by the numerical designation 2 to 9. These eight references are specially prepared by blending varying proportions of wool dyed with CI Mordant Blue 1 (Colour Index, third edition, 43830) and wool dyed with CI Solubilized Vat Blue 8 (Colour Index, third edition, 73801), so that each higher-numbered reference is approximately twice as fast as the preceding reference.

4.1.3 Humidity test control

Effective humidity is defined as the combination of air and surface temperatures and air relative humidity which governs the moisture content of the surface of the test specimen during exposure. The effective humidity can be measured only by determining the colour fastness of a specific humidity test control.

For the purposes of this part of ISO 105, the humidity test control is a red azoic dyed cotton cloth.

This control has been calibrated by exposing it facing south in several Western European locations at different times of the year, the exposures being made together with the references in sealed vessels containing air maintained at constant humidities between 0 and 100 %; the results did not vary greatly and the mean values are shown in figure 1.

When this control was exposed under the conditions specified in ISO 105-B01 in temperate zones, its colour fastness was found to be, on average, 5.

Table 1 — Dyes for blue wool references 1 to 8

Reference	Dye (Colour Index designation) [1]
1	CI Acid Blue 104
2	CI Acid Blue 109
3	CI Acid Blue 83
4	CI Acid Blue 121
5	CI Acid Blue 47
6	CI Acid Blue 23
7	CI Solubilized Vat Blue 5
8	CI Solubilized Vat Blue 8

1) The Colour Index (third edition) is published by the Society of Dyers and Colourists, P.O. Box 244, Perkin House, 82 Grattan Road, Bradford BD1 2JB, West Yorks, UK, and by the American Association of Textile Chemists and Colorists, P.O. Box 12215, Research Triangle Park, NC 27709-2215, USA.

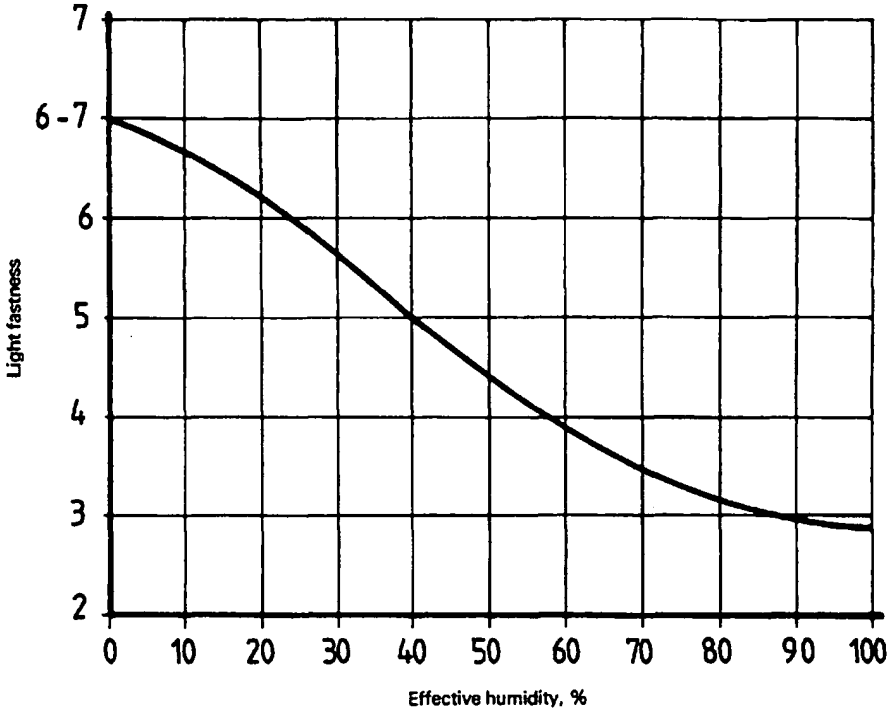


Figure 1 — Mean values obtained from humidity test control exposures

4.2 Apparatus

4.2.1 Xenon arc lamp apparatus, either air-cooled or water-cooled.

The specimens and the references are exposed in one of the two types of apparatus (see 4.2.1.1 and 4.2.1.2). The variation in light intensity over the area covered by the specimens and references shall not exceed ± 10 % of the mean. The recommended level of irradiance (radiant power per unit area) measured by a radiometer (4.2.6) is 42 W/m^2 × wavelength, in nanometres, at 300 nm to 400 nm, equivalent in cases of water-cooled xenon arc apparatus to 1,1 W/m^2 × wavelength, in nanometres, at 420 nm.

The distances from the surface of the specimen and from the surfaces of the references to the lamp shall be the same.

4.2.1.1 Air-cooled xenon arc lamp apparatus (see annex A), consisting of the following elements:

NOTE 2 For exposure conditions preferable for use in Europe, see 6.1.

a) **Light source**, in a well-ventilated exposure chamber.

The light source shall consist of a xenon arc lamp of correlated colour temperature 5 500 K to 6 500 K, the size of which will depend on the type of apparatus used.

b) **Light filter**, placed between the light source and the specimens and references so that the ultraviolet spectrum is steadily reduced.

The transmission of the filter system used shall be at least 90 % between 380 nm and 750 nm, falling to 0 between 310 nm and 320 nm.

c) **Heat filter**, placed between the light source and the specimens and references so that the amount of infrared (IR) radiation contained in the xenon arc spectrum is steadily reduced (see A.1.1 and A.2.2).

If a glass or water filter is used to eliminate excess infrared radiation so as to meet the temperature conditions specified in 4.2, frequent cleaning shall be carried out to avoid unwanted filtering caused by dirt (see B.1.4).

4.2.1.2 Water-cooled xenon arc lamp apparatus *(see annex B), consisting of the following* elements:

a) **Light source**, in a well-ventilated exposure chamber.

The light source shall consist of a xenon arc lamp of correlated colour temperature 5 500 K to 6 500 K, the size of which will depend on the type of apparatus used.

b) **Light filter**, consisting of inner and outer filter glass containing and directing the flow of cooling water, placed between the light source and the specimens and references so that the ultraviolet spectrum and a part of the infrared spectrum are steadily reduced.

For exposure conditions preferable for use in Europe [see 6.1 a) and 6.1 b)]: inner and outer IR filter glass and a lantern, equipped with European window glass. The transmission of the filter system used shall be at least 90 % between 380 nm and 750 nm, falling to 0 between 310 nm and 320 nm.

For exposure conditions preferable for use in America (see 6.2): an inner filter of Pyrex (borosilicate) glass and an outer filter of clear (soda lime) glass are used so that the irradiation at the specimen has a lower spectral cut-off approximately equal to that of window glass (see B.1.2).

c) **Heat filter**, comprising grade 3 water (see ISO 3696) circulating through the lamp assembly between the inner and outer filter glass, cooled by passing through a heat-exchange unit (see B.1.4).

4.2.2 Opaque cardboard or other thin opaque material, for example thin sheet aluminium or cardboard covered with aluminium foil, for partial covering of samples and references.

4.2.3 Temperature sensor, either a black-panel thermometer (BPT) or black-standard thermometer (BST).

4.2.3.1 The black-panel thermometer (BPT) shall consist of a metal panel at least 45 mm × 100 mm whose temperature is measured with a thermometer or a thermocouple whose sensitive portion is located in the centre of and in good contact with the panel.

The side of the panel facing the light source shall be black with a reflectance of less than 5 % throughout the spectrum of light reaching the specimen; the side of the panel not facing the light source shall be thermally insulated (see also B.1.5).

4.2.3.2 The black-standard thermometer (BST) shall consist of a plane of stainless steel plate measuring about 70 mm × 30 mm and with a thickness of about 0,5 mm, whose temperature is measured by a thermal resistor, with good heat-conducting properties, fitted to the reverse side. The metal plate is fixed to a plastics plate so that it is thermally insulated. It is coated with a black layer which has an absorption of at least 95 %, even in the infrared range.

4.2.4 Grey scale for assessing change in colour, complying with ISO 105-A02.

4.2.5 Colour-matching lamp, in accordance with CIE Publication No. 51, for assessing change in whiteness.

4.2.6 Radiometer, (when available/specified) for measuring irradiance and radiant exposure in the wavelength range 300 nm to 400 nm or at a specific wavelength (e.g. 420 nm) (see A.1.7 and B.1.8).

Since irradiance at the test specimen face can vary as a function of lamp intensity and lamp-to-specimen distance, a monitoring radiometer may be used to control uniformity of exposure. The radiometer permits exposure to an established level of irradiance (radiant energy flux per unit area) at a point in the plane of the specimen rack.

5 Test specimens

The size of the test specimen will depend on the number of specimens to be tested and on the shape and dimensions of the specimen holders supplied with the apparatus.

5.1 In apparatus of the air-cooled type (4.2.1.1), an area of the textile not less than 45 mm × 10 mm is usually used when several periods of exposure are made side by side on the same specimen, which is the preferred practice. The specimen may be a strip of cloth, yarn wound close together on a card or laid parallel and fastened on a card, or a mat of fibres combed and compressed to give a uniform surface and fastened on a card. Each exposed and unexposed area shall be not less than 10 mm × 8 mm.

5.2 To facilitate handling, the specimen or specimens to be tested and similar strips of the references may be mounted on one or more cards as indicated in figure 2 or 3.

5.3 In apparatus of the water-cooled type, specimen holders are fitted to take specimens of approximately 70 mm × 120 mm. When desired, specimens of different sizes to fit alternative sizes of specimen holder may be used. References shall be exposed on a white cardboard backing. Specimens may also be mounted on white cardboard if desired.

5.4 The covers (4.2.2) shall make close contact with the surface of the unexposed areas of the specimens and the references, in order to give a sharp line of demarcation between exposed and unexposed areas, but shall not compress the specimen unnecessarily.

5.5 The specimens to be tested and the blue wool references shall be of equal size and shape in order to avoid errors in assessment due to overrating the visual contrast between exposed and unexposed parts on a larger pattern as against a narrower reference (see 8.4).

5.6 When testing pile fabrics, the references shall be arranged so that they are the same distance from the light source as the surface of the pile fabrics. This can be achieved for example by using pieces of cardboard as underlay. Covers for the unexposed portions shall avoid surface compression.

Pile fabrics, such as carpets, which have fibres that may shift position or texture which may make evaluation of small areas difficult, shall be tested with an exposed area not less than 50 mm × 40 mm and preferably larger.

6 Exposure conditions

6.1 Conditions preferable for use in Europe

a) **Normal conditions** (temperate zone): moderate effective humidity (see 4.1.3); colour fastness of the humidity test control: 5; maximum black-standard temperature: 50 °C (see 4.2.3.2).

b) **Extreme conditions**: For testing the sensitivity of specimens to different humidity during irradiation, the following extreme conditions are useful:

1) low effective humidity:

— colour fastness of the humidity test control: 6-7;

— maximum black-standard temperature: 65 °C;

2) high effective humidity:

— colour fastness of the humidity test control: 3;

— maximum black-standard temperature 45 °C.

NOTE 3 Measurements with the black-panel thermometer (BPT) (4.2.3.1) yield a temperature 5 °C lower than the more modern black-standard thermometer (BST) (4.2.3.2).

6.2 Conditions preferable for use in America

Black-panel temperature (63 ± 1) °C (see 4.2.3.1); the controls of the apparatus are adjusted so that the relative humidity of the air in the test chamber is (30 ± 5) %. Effective humidity: low. Colour fastness of the humidity test control: 6-7.

These conditions shall be controlled by continuous operation of a blower, with concomitant measuring and thermostatic control of the temperature of a constant volume of air whose relative humidity is increased by adding moisture to the air as it passes through the conditioning chamber by means of an electrically operated atomizer.

7 Procedure

7.1 Adjustment of the humidity (see clause 6)

7.1.1 Check that the apparatus is in good running order and that it is equipped with a clean xenon burner tube. (Follow the manufacturer's directions and see annexes A and B.)

7.1.2 Place a portion of the humidity test control (4.1.3) measuring not less than 45 mm × 10 mm, together with the blue wool references (4.1.1 or 4.1.2), on a card, if possible in the middle area of the specimen holder (see 5.3).

7.1.3 Place the filled specimen holders on the specimen rack of the apparatus, with the holders supported both top and bottom and in proper vertical alignment. Completely fill all remaining spaces in the specimen rack with holders containing white cardboard.

7.1.4 Operate the apparatus with the light on continuously until a test is completed unless the lamp requires cleaning or the burner, outer filter or inner filter requires changing because it has reached the maximum number of hours of recommended usage.

7.1.5 Expose the partially covered strip of the humidity test control and the references simultaneously until a contrast between the exposed and unexposed portions equal to grade 4 on the grey scale is produced on the humidity test control.

7.1.6 Assess the colour fastness of the humidity test control at this stage and, if necessary, adjust the controls on the apparatus to give the selected exposure conditions. Check daily and when necessary readjust the controls to maintain the specified black-panel (black-standard) temperature and humidity.

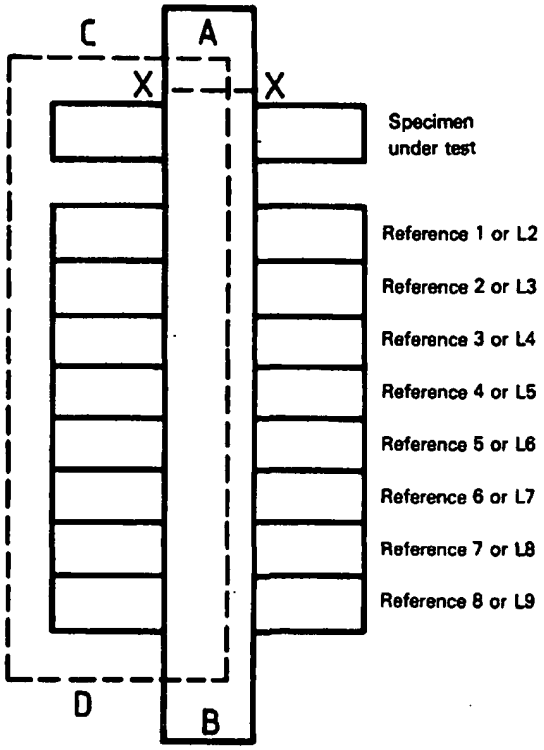
7.2 Exposure methods

Expose the specimen (or group of specimens) and the references simultaneously, under the desired conditions, in such a manner and for such a time as is necessary to evaluate fully the colour fastness of each specimen relative to that of the references, by progressively covering both the specimens and the exposed references during the test (using either method 1 or method 2). Covering arrangements other than those described, for instance covering specimens and references on both ends and exposing the middle third or half, are also allowed.

7.2.1 Method 1

This method is considered most exact and should be used in cases of dispute over the numerical rating. The basic feature is the control of the exposure period by inspection of the *specimen*, and therefore only one set of blue wool references is required for each specimen under test.

7.2.1.1 Arrange the test specimen and the references as shown in figure 2 with an opaque cover AB across the middle one-third of the specimen and references. Expose to the xenon arc light under the conditions enumerated in 7.1. Follow the effect of light by removing the cover and inspecting the specimen frequently. When a change in the test specimen can be perceived equal to grey scale 4-5, note the number of the blue wool reference showing a similar change. (This is a preliminary assessment of colour fastness.)



AB: Opaque cover; may be hinged at X-X so that it can be lifted and returned to the same place over the specimen and references

CD: Second cover

Figure 2 — Mounting of specimen and references for exposure method 1

If there is a possibility of the sample being photochromic, then at this stage the test for photochromism shall be applied additionally (see ISO 105-B05).

For all specimens except for white (bleached or optically brightened) specimens, continue the procedure as described in 7.2.1.2 to 7.2.1.4. For optically brightened textiles, continue with the procedure as described in 7.2.1.5.

7.2.1.2 Continue to expose until the contrast between the exposed and the unexposed portions of the specimen is equal to grey scale grade 4. Cover the left-hand one-third of the specimen and references with an additional opaque cover (CD in figure 2).

7.2.1.3 Continue to expose until the contrast between the fully exposed and unexposed portions of the test specimen is equal to grey scale grade 3.

7.2.1.4 If reference 7 or L7 fades to a contrast equal to grey scale grade 4 before the test specimen does, the exposure is terminated at this stage. When a specimen has a colour fastness equal to or greater than 7 or L7, it would require unduly long exposure to produce a contrast equal to grey scale grade 3; moreover, this contrast would be impossible to obtain when the colour fastness is 8 or L9. Assessments in the region of 7-8 or L7-L8 are made, therefore, when the contrast produced on reference 7 or L7 is equal to grey scale 4, the time required to produce this contrast being long enough to eliminate any error which might result from inadequate exposure.

7.2.1.5 For white (bleached or optically brightened) textiles, continue to expose until the contrast between the exposed and unexposed portions of the specimen is equal to grey scale grade 4.

7.2.2 Method 2

7.2.2.1 This method should be used when a large number of specimens have to be tested simultaneously. The basic feature is the control of the exposure periods by inspection of the *references*, which allows a number of specimens differing in colour fastness to be tested against a single set of references, thus conserving supplies.

7.2.2.2 Arrange the test specimens and the references as shown in figure 3 with the cover AB covering one-quarter of the total length of each specimen and reference. Expose under the conditions enumerated in 7.1.1 and 7.1.2. Follow the effect of light by lifting the cover AB periodically and inspecting the references. When a change in reference 3 or L2 can be perceived equal to grey scale grade 4-5, inspect the specimens and rate their colour fastness by comparing any change that has occurred with the changes that have occurred in references 1, 2 and 3 or L2. (This is a preliminary assessment of colour fastness.)

If there is a possibility of the sample being photochromic, then at this stage the test for photochromism shall be applied additionally (see ISO 105-B05).

7.2.2.3 Replace the cover AB in exactly the same position and continue to expose the specimens until a change in reference 4 or L3 can be perceived equal to grey scale grade 4-5; at this point fix an additional cover, CD, in the position shown in figure 3), overlapping the first cover, AB.

7.2.2.4 Continue to expose the specimens until a change in reference 6 or L5 can be perceived equal to grey scale 4-5, then fix the final cover, EF, in the position shown in figure 3, the other two covers remaining in position.

7.2.2.5 Expose until either

a) a contrast is produced on reference 7 or L7 equal to the contrast illustrated by grey scale grade 4; or

b) a contrast equal to grey scale grade 3 has been produced on the most resistant specimen; or

c) for white (bleached or optically brightened) textiles, a contrast equal to grey scale grade 4 has been produced on the most resistant specimen;

whichever occurs first.

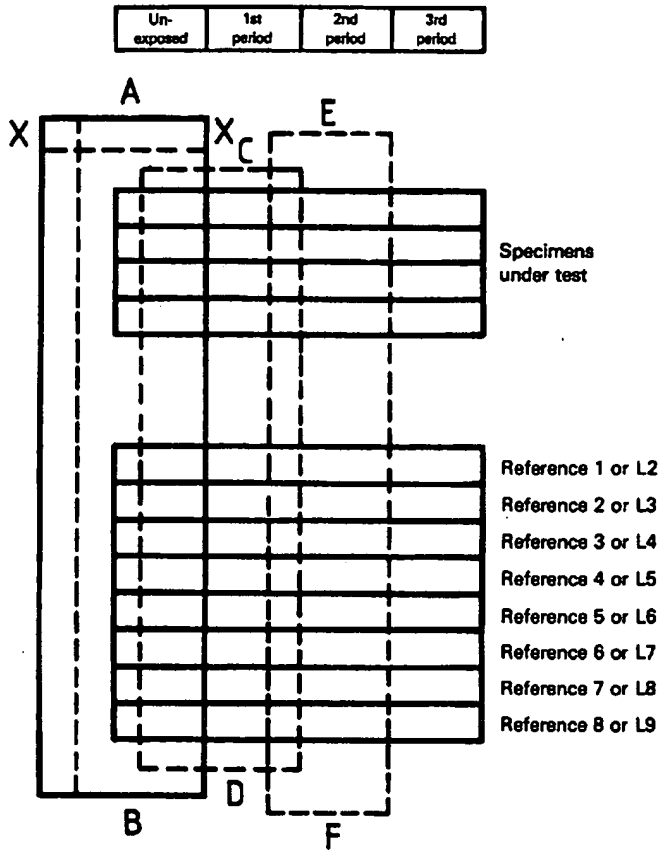
NOTE 4 The contrast referred to in a) and b) may occur before the fading defined in 7.2.2.3 or 7.2.2.4 has taken place.

7.2.3 Method 3

Where the test is to be used to check conformity with a performance specification, it is permissible to expose the specimens with two blue wool references only: that specified as minimum and the one below it. Continue exposure until grey scale grade 4 and grey scale grade 3 contrasts have been produced on separate areas of the minimum blue wool reference. For white (bleached or optically brightened) textiles, continue exposure until a grey scale grade 4 contrast has been produced between separate areas of the minimum blue wool reference.

7.2.4 Method 4

Where the test is to be used to check conformity with an agreed-upon reference sample, it is permissible to expose the specimens with the reference sample only. Continue exposure until grey scale grade 4 and/or grey scale grade 3 contrasts have been produced on the reference sample. For white (bleached or optically brightened) textiles, continue exposure until a grey scale grade 4 contrast has been produced on the reference sample.



AB: Opaque cover; may be hinged at X-X so that it can be lifted and returned to the same place over the specimen and references

CD: Second cover

EF: Third cover

Figure 3 — Mounting of specimens and references for exposure method 2

7.2.5 Method 5

Where the test is to be used to check conformity to agreed-upon radiant energy levels, it is permissible to expose the specimens alone or with blue wool references. The specimens should be exposed until the specified amount of radiant energy is reached then removed, together with the blue wool references, and evaluated in accordance with 8.9.

8 Assessment of colour fastness

8.1 The final assessment given as numerical ratings, is based on contrasts equal to grey scale grade 3 between exposed and unexposed portions of the test specimen. For white (bleached or optically brightened) textiles, the final assessment in numerical ratings is based on a contrast equal to grey scale grade 4 between exposed and unexposed portions of the specimen.

8.2 Remove all the covers, thus revealing on test specimens and references two or three areas, depending on the method used, which have been exposed for different times, together with at least one area which has not been exposed to light. Compare the changes in the specimen with the relevant changes in the references under suitable illumination (see ISO 105-A01:1994, clause 14).

For white (bleached or optically brightened) textiles, the use of artificial daylight produced by a colour-matching lamp (4.2.5) is recommended and is necessary in cases of dispute, unless otherwise agreed.

The colour fastness of the specimen is the number of the reference which shows similar changes in colour (visual contrast between exposed and unexposed parts of the specimen). If the specimen shows changes in colour which are nearer to the imaginary reference midway between any two consecutive references, an intermediate rating, for example 3-4 or L2-L3, shall be given.

If different assessments are obtained at the different degrees of contrast, the colour fastness of the specimen is taken as the arithmetic mean of these, expressed to the nearest half or whole grade. When three areas are being rated, take the mean of the contrasts closest to grey scale grades 4 and 3. Assessments, however, shall be confined to whole or midway ratings only. When the arithmetic mean gives a quarter or three-quarters rating, the assessment is defined as the next higher half or whole grade.

However, to avoid a misrating of the colour fastness of the specimen due to its photochromism, the specimen should be allowed to condition in the dark at room temperature for 24 h before assessing the colour fastness (see ISO 105-B05).

8.3 If the colour of the specimens is more fugitive than that of reference 1 or L2, a rating of 1 or L2 is given.

8.4 Comparison of the changes in the test specimen with changes in the references may be facilitated by surrounding the specimen with a mask of neutral grey colour approximately midway between the lighter chips in grades 1 and 2 (approximately Munsell N5), and surrounding the references in turn with a similar mask of equal aperture.

8.5 If the colour fastness is equal to or higher than 4 or L3, preliminary assessment based on the contrast equal to grey scale grade 4-5 (see 7.2.1.2 and 7.2.2.2) becomes significant; if this preliminary assessment is 3 or lower or L2, it shall be included in the rating in brackets. For example, a rating of 6(3) or L5(L2) indicates that the specimen changes very slightly in the test when reference 3 or L2 just begins to fade, but that on continuing the exposure the resistance to light is equal to that of reference 6 or L5.

8.6 If the specimen is photochromic, the colour fastness rating shall include a P bracketed with the rating obtained from the test for photochromism, for example 6(P3-4) (see ISO 105-B05).

8.7 The term "change in colour" includes change in hue, chroma, lightness, or any combination of these characteristics of colour (see ISO 105-A02:1993, subclause 2.6).

8.8 Exposures based on a performance reference (see 7.2.3) or together with an agreed-upon reference sample (see 7.2.4) shall be assessed by comparison of the colour changes of the test specimen and the references. If the specimen shows no greater change in colour than the performance reference or the reference sample, the colour fastness shall be classified "satisfactory"; if the specimen shows a greater change in colour than the performance reference or the reference sample, the colour fastness shall be classified "unsatisfactory".

8.9 The assessment of the colour fastness according to method 5 (see 7.2.5) is performed either by comparison with the grey scale for assessing change in colour, in accordance with ISO 105-A02, or by comparison of the change in colour of the sample with that of the references.

9 Test report

The test report shall include the information:

a) the number and year of publication of this part of ISO 105, i.e ISO 105-B02:1994;

b) all details necessary for the identification of the sample tested;

c) for method 1 or method 2:

Report the numerical rating for the colour fastness to light. The colour fastness rating shall be expressed either:

1) by the figure alone (when using the blue wool references designated 1 to 8); or

2) together with the prefix L (when using the blue wool references designated L2 to L9).

If this rating is equal to or higher than 4 or L3 and the preliminary assessment is equal to or lower than 3 or L2, report the latter figure in brackets.

If the specimen is photochromic, the colour fastness shall be followed by a P bracketed together with the grey scale rating obtained from the test for photochromism, for example 6(P3-4).

d) for method 3 or method 4:

Report the classification "satisfactory" or "unsatisfactory", together with the performance reference or the reference sample used.

e) for method 5:

Together with the specified amount of radiant energy, report the numerical rating for colour fastness to light. The rating shall be expressed either:

1) by the figure alone (when using the blue wool references designated 1 to 8); or

2) together with the prefix L (when using the blue wool references L2 to L9);

3) if no blue wool references are used, by the rating of the colour change assessed by comparison with the grey scale in accordance with ISO 105-A02 or by colour measurement in accordance with ISO 105-A05.

f) The apparatus used, the method, the exposure conditions and the assessment conditions.

Annex A
(normative)

Apparatus for determining colour fastness with air-cooled xenon arc lamps

A.1 Description and conditions of use

A.1.1 The test apparatus (see also A.2) is equipped with an air-cooled xenon arc lamp as the source of radiation. The two models of apparatus use xenon arc lamps with a wattage of 1 500 W or 4 500 W. The xenon arc lamp is surrounded by a filter system consisting of a quartz inner cylinder, an additional lamp-chimney of seven heat filters and an outer cylinder of ultraviolet filter glass. With this filtering device the requirements given in 4.2.1 are satisfied.

A.1.2 The space between the xenon arc lamp and the filtering device is cooled by a current of air. This cooling air should be discharged into the open atmosphere.

A.1.3 The decrease in intensity of air-cooled xenon arc lamps due to ageing is small. After 1 500 h of use, the radiation flux drops to approximately 90 % and replacement of the lamps is recommended.

The change in the transmission properties of the heat filter due to ageing can be eliminated almost entirely by replacing the oldest filter in rotation after 500 h.

A.1.4 Slot-in specimen holders are mounted on a revolving rack and at a given distance in circular fashion around the vertical lamp unit. The rack rotates at 500 r/min. After each revolution of the rack, the specimen holders are turned 180° about their longitudinal axis.

The surface area of specimen radiated at any one time is 450 cm^2 in the case of the test apparatus with a 1 500 W xenon arc lamp and 1 800 cm^2 with a 4 500 W xenon arc lamp.

A.1.5 An air current generated with a blower is directed through the test chamber and over the surface of the specimen. In the case of the apparatus with a 1 500 W xenon arc lamp, the test chamber is air-conditioned by adding moisture to the air via spray jets or by means of an ultrasonic humidification device, while in the case of the apparatus with a 4 500 W xenon arc lamp, water is atomized very finely via an aerosol device and added to the air current. Measurement and control of the relative humidity in the test chamber is carried out either by a contact hygrometer or by electronic means.

A.1.6 The exposure conditions, relative to effective humidity, specified in this part of ISO 105 are achieved in the test apparatus by regulating the relative humidity of the air within certain values, which are given in the instructions for using the apparatus.

The test chamber temperature and black-panel temperature can be influenced by appropriately throttling the air supply. In the case of the apparatus with a 4 500 W xenon arc lamp, it is possible, by connecting to a heating and/or cooling device, to control the test chamber temperature over a wide range.

A.1.7 A monitoring radiometer mounted on the test specimen area can be useful in this method. A radiometer employing a broad bandpass filter restricting measurement to the ultraviolet spectral region between 300 nm and 400 nm has been used satisfactorily.

Filter radiometers capable of integrating irradiance with respect to time are satisfactory.

The calibration of the radiometer shall be certified by the manufacturer for a specified time interval when used in accordance with this part of ISO 105.

A.2 Use of an alternative apparatus

A.2.1 The description and conditions of use of this alternative apparatus are similar to those given in A.1, except for the changes given in A.2.2 and A.2.3.

A.2.2 There are three different models of this alternative apparatus, with a wattage of 1 500 W, 2 500 W or 4 500 W. The xenon arc lamp is surrounded by a filter system consisting of an outer cylinder of ultraviolet-filter glass and a lamp-chimney of six heat filters.

A.2.3 After each revolution of the rack, the specimen holders are either turned 180° around their longitudinal axis or maintained facing the xenon arc lamp for the 1 500 W and 4 500 W models. The specimen holders always face the xenon arc lamp for the 2 500 W model.

Annex B
(normative)

Apparatus for determining colour fastness with water-cooled xenon arc lamps

B.1 Description and conditions of use

B.1.1 The test apparatus incorporates a 6 500 W water-cooled xenon long-arc lamp as the source of radiation. While all of the xenon arc lamps employed are of the same general type, different-sized lamps operating in different wattage ranges are employed in several sizes and types of apparatus. In each of the various models, the diameter of the specimen rack, lamp size and lamp wattage can be varied so that, when the specimens are exposed in the holders, the irradiance at the face of the specimens is at the appropriate level.

B.1.2 The xenon lamp consists of a xenon burner tube, inner glass filter, outer glass filter, and the necessary accessories. In some cases, additional glass filters may be installed to reduce infrared radiation. For colour fastness tests according to 6.2, a borosilicate glass inner filter and a soda lime glass outer filter are used so that the irradiation at the specimen has a lower spectral cutoff value approximately equal to that of window glass. When operating the apparatus according to 6.2, outer filters shall be discarded after 2 000 h of use and inner filters after 400 h of use. For colour fastness tests according to 6.1 a) and 6.1 b), a lantern equipped with European window glass is used in addition to an infrared inner and outer filter combination. Because of the drop in intensity with continued use, xenon burners shall be discarded when 1,1 $W/(m^2 \cdot nm)$ at 420 nm is no longer achievable by automatic control.

B.1.3 All xenon arc exposure apparatus is equipped with suitable starters, reactance transformers and indicating and control equipment for either manually or automatically controlling the wattage of the lamp. In manually controlled units, the wattage of the lamp may require periodic adjustment to maintain 1,1 $W/(m^2 \cdot nm)$ at 420 nm.

B.1.4 To cool the lamp, grade 3 water is circulated through the lamp assembly at an approximate minimum flow rate of 380 l/h, and is purified further by the use of a mixed bed deionizer just ahead of the lamp. The recirculated lamp water is cooled without contamination by the use of a heat exchanger unit employing either tap water or a refrigerant as the heat transfer medium.

B.1.5 The exposure apparatus is enclosed in an insulated cabinet to minimize the effects of variation in room temperature. A ventilating system provides a varying volume of air through the test chamber and over the test specimens. The temperature of the air and the black standard thermometer are automatically controlled by varying the volumes of warm air circulated from the test chamber mixed with cooler room air. Moisture in the amount required to maintain the specified relative humidity of the exit air from the test chamber as measured by wet-and-dry bulb temperatures may be added to the air system as it passes through the air-conditioning chamber in the base of the instrument.

B.1.6 A cylindrical vertical or inclined frame or rack supporting the specimen holders is rotated at 1 r/min around the lamp, which is located centrally with respect to the specimen rack so that the effective arc is centred both horizontally and vertically relative to the exposure area of the specimen holders.

B.1.7 Apparatus for use in this method are equipped with timing units for controlling the length of exposure. Some apparatus are also equipped with a light monitor designed to switch off the apparatus as soon as a given radiant exposure has been achieved.

B.1.8 Apparatus for use in this method may be equipped with a monitoring/controlling radiometer for controlling the length of exposure. A radiometer employing a narrow bandpass interference filter restricting measurement to the UV spectral region has been used satisfactorily. It consists of a sensor employing a photodetector and interference filter with central wavelength tolerance no greater than 2 nm, half-bandwidth no greater than 20 nm.

Single or multiple filter radiometers capable of measuring, recording, controlling and/or integrating irradiance with respect to time are satisfactory.

For monitors designed to automatically maintain a constant level of irradiance, exposures of equal time should provide equivalent radiance exposure, which may be calculated by the following formula:

$$H = E3{,}6t$$

where

H is the radiance exposure, expressed in kilojoules per square metre;

E is the irradiance, expressed in watts per square metre (joules per square metre per second);

t is the time passed, in hours;

3,6 is a conversion constant (kiloseconds to hours).

Single filter radiometers equipped with a presetable countdown integrator, calibrated in kilojoules per square metre, designed for use with the exposure apparatus, can terminate the test when the specimens have received the preset level of radiance exposure.

The radiometer shall have means for checking the calibration provided by the manufacturer, or calibration shall be certified by the manufacturer for a specified time interval when used in the manner described herein.

B.2 Use of an alternative apparatus

B.2.1 The description and conditions of use of this alternative apparatus are similar to those given in B.1, except for the different sizes in operating lamps listed in B.2.2.

B.2.2 In addition to the model listed in B.1.1, alternative models of the apparatus are available with lamps of 2 500 W, 4 500 W and 6 500 W.

Annex C
(informative)

General information on colour fastness to light

C.1 When in use, textiles are usually exposed to light. Light tends to destroy colouring matters and the result is the well-known defect of "fading", whereby coloured materials change colour—usually becoming paler and duller. Dyes used in the textile industry vary enormously in their resistance to light and it is obvious that there must be some method of measuring their fastness. The substrate also influences the colour fastness of a dye to light.

This part of ISO 105 cannot satisfy completely all the interested parties (who range from dye manufacturers and the textile industry to wholesale and retail traders and the general public) without becoming technically involved and possibly difficult to understand by many who have a direct interest in its application.

C.2 The following non-technical description of a test for colour fastness to light has been prepared for the benefit of those who find the detailed technicalities of this part of ISO 105 difficult to understand. The method is to expose the pattern being tested and to expose also, at the same time and under the same conditions, a series of colour fastness references which are pieces of wool cloth dyed with blue dyes of different degrees of fastness. When the pattern has faded sufficiently, it is compared with the references and if it has behaved, for instance, like reference 4[3], then its colour fastness is said to be 4.

C.3 The colour fastness references should cover a wide range, since some patterns fade noticeably after exposure for 2 h or 3 h to bright summer sunshine, although others may withstand long exposure without change, the dyes in fact outliving the material to which they have been applied. Eight references have been chosen, reference 1 being the most fugitive and reference 8 the most resistant. If it takes a certain length of time for reference 4 to fade under certain conditions, then the same amount of fading will occur on reference 3 in approximately half that time, or on reference 5 in approximately twice that time, provided that the conditions are the same.

C.4 It is necessary to ensure that different people testing the same material will fade it to the same extent before assessment against the simultaneously faded reference. The ultimate users of dyed material differ widely in what they consider to be "faded articles" and therefore patterns under test are faded to two different degrees which adequately cover most opinions and make assessment more reliable. These required degrees of fading are defined by reference to a collection of "grey scale" reference contrasts (grey scale 5 equals no contrast, grey scale 1 equals large contrast). Thus the use of the grey scale enables fading to be taken to defined extents, and the blue wool cloths enable the colour fastness to be rated.

This general principle of assessing on the basis of moderate and severe fading is complicated, however, by the fact that some patterns on exposure undergo a slight change very rapidly indeed but do not change further for a long time. These slight changes are such that under normal conditions of use they would seldom be observed, but in certain cases they become important, as the following example shows.

A retailer has a length of curtain fabric in his window and on it is a cardboard ticket indicating the price. After a few days the ticket is removed and careful examination reveals the place where it has been resting because the surrounding cloth has changed shade slightly on exposure to light. Some of this curtain material is exposed so as to produce a moderate degree of fading and it is found that reference 7 has faded to the same extent; the general colour fastness of the fabric is therefore 7.

The important factor about this slight change in shade is that it can only be detected when there is a sharp boundary between the exposed and unexposed areas, and these conditions rarely occur during normal use. The magnitude of this slight change would be given as an additional assessment in brackets. Thus a rating for a test could be 7(2), indicating a slight initial

3) The designations of the colour fastness references referred to here are those of the European set (see 4.1.1). The principles explained are equally valid for the American set (see 4.1.2).

change equivalent to the first perceptible fade of reference 2, but otherwise a high colour fastness of 7.

C.5 A further unusual colour change is also catered for, namely photochromism. This effect is shown when a dye changes colour rapidly on exposure to strong light but on removal to a dark place the original colour returns more or less completely. The extent of photochromism is determined by the special test described in ISO 105-B05, and is shown in the rating by a number following the letter P within brackets; for example 6(P2) means a photochromic effect equal to a grey scale 2 contrast but permanent fading equal to that of reference 6.

C.6 Finally, there are many patterns which change hue on prolonged exposure to light; for example, a yellow may become brown, or a purple may become blue. In the past there have been many arguments as to whether such patterns could be said to have faded or not. The technique used in ISO 105-B01 to ISO 105-B05 is unambiguous on this point; it is visual contrast on exposure which is being measured, whether it be loss of colour or change in hue; in the latter case, however, the kind of change is included in the assessments. For example, consider two green patterns which, on exposure, change in appearance at the same rate as reference 5; one becomes increasingly pale while the other becomes first a greenish blue and finally a pure blue. The former would be rated "5" and the latter "5 bluer". In this instance also, the technique used in ISO 105-B01 to ISO 105-B05 tries to present as complete a picture of the behaviour of a pattern on exposure as is possible without becoming excessively complicated.

ICS 59.080.10

Descriptors: textiles, dyes, tests, daylight tests, artificial light tests, determination, colour fastness.

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AMENDMENT 1
1998-08-01

Textiles — Tests for colour fastness —

Part B02:
Colour fastness to artificial light: Xenon arc fading lamp test

AMENDMENT 1

Textiles — Essais de solidité des teintures —

Partie B02: Solidité des teintures à la lumière artificielle: Lampe à arc au xénon

AMENDEMENT 1

Reference number
ISO 105-B02:1994/Amd.1:1998(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Amendment 1 to ISO 105-B02:1994 was prepared by Technical Committee ISO/TC 38, *Textiles*, Subcommittee SC 1, *Tests for coloured textiles and colorants*.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

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Textiles — Tests for colour fastness —

Part B02:
Colour fastness to artificial light: Xenon arc fading lamp test

AMENDMENT 1

Page 2, 4.1 Reference materials

After first sentence insert:

The relationship between references 1 to 8 and L2 to L9 as shown with the method are approximate. Results from testing which use reference standards from both sources should be compared only with the knowledge that fading characteristics may differ.

4.1.2

Insert the following as a second paragraph of 4.1.2:

Data in annex D are presented to illustrate the relationship of each of the blue wool references on exposure to fixed amounts of radiant energy. A detailed summary of these test results is found in document reference number ISO/TC38/SC1/N993.

Page 5

Under 6.1 insert the following:

For these conditions, use the references 1 to 8 specified in 4.1.1.

Under 6.2 insert the following:

For these conditions, use the references L2 to L9 specified in 4.1.2.

Page 6, 7.2.1 Method 1

7.2.1.1 Delete the existing text and substitute:

7.2.1.1 Arrange the specimen to be tested and the references as shown in figure 2 with an opaque cover AB across the middle one-third of the specimen and references. Expose to the xenon arc light under the conditions enumerated in 6.1 or 6.2. Follow the effect of exposure by removing the cover and inspecting the

specimen frequently until the contrast between the exposed and the unexposed portions of the specimen is equal to grey scale grade 4. Cover the left-hand one-third of the specimen and references with an additional opaque cover (CD in figure 2). At this stage attention should be given to the possibility of photochromism (see ISO 105-B05). For white (bleached or optically brightened) textiles, continue as described in 7.2.1.4.

7.2.1.2 Delete the existing text (included in 7.2.1.1).

7.2.1.3 Renumber as 7.2.1.2.

Page 7

7.2.1.4 Renumber as 7.2.1.3.

7.2.1.5 Renumber as 7.2.1.4.

7.2.2 Method 2

7.2.2.2 to 7.2.2.4 Delete the existing text and substitute:

7.2.2.2 Arrange the specimens to be tested and the references as shown in figure 3, with cover AB covering one-fifth to one-quarter of the total length of each specimen and reference. Expose to light under the conditions enumerated in 6.1 or 6.2. Follow the effect of exposure by lifting cover AB periodically and inspecting the references. When a change in reference 2 can be perceived equal to grey scale grade 3 or L2 to 4 inspect the specimens and rate their colour fastness by comparing any change that has occurred with the changes that have occurred in references 1, 2 and 3 or L2. (This is a preliminary assessment of colour fastness.) At this stage attention shall be given to the possibility of photochromism (see ISO 105-B05).

7.2.2.3 Replace the cover AB in exactly the same position and continue to expose until a change in reference 4 or L3 can be perceived equal to grey scale grade 4; at this point fix an additional cover CD in the position shown in figure 3, overlapping the cover AB.

7.2.2.4 Continue to expose until a change in colour in reference 6 or L4 can be perceived equal to grey scale grade 4; then fix the final cover EF in the position shown in figure 3, the other covers remaining in position.

Page 9, 8.5

Delete the existing text and substitute:

8.5 If the colour fastness is equal to or higher than 4 or L3, any preliminary assessment (see 7.2.2.2) becomes significant; if this preliminary assessment is 3 or L2, it shall be included in the rating in brackets. For example, a rating of 6(3) indicates that the specimen changes very slightly in the test when reference 3 just begins to fade, but that on continuing the exposure the resistance to light is equal to that of reference 6.

Page 11, A.1.4

In line 4 delete "500 r/min"; and insert "5 r/min".

New page

Annex D
(informative)

Light exposure equivalents for blue wool lightfastness references L2 to L9 [1)]

Blue wool reference	Xenon only	
	420 nm kJ/m²	300 nm to 400 nm kJ/m²
L2	21	864
L3	43	1 728
L4	85 [2)]	3 456
L5	170	6 912
L6	340 [2)]	13 824
L7	680	27 648
L8	1 360	55 296
L9	2 720	110 592

1) For colour change of step 4 on the grey scale for colour changes.

2) Verified by experiment; all other values are calculated.

ICS 59.080.01

Descriptors: textiles, dyes, tests, daylight tests, artificial light tests, determination, colour fastness.

Price based on 3 pages